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(71) Applicant: MONSANTO COMPANY [US/US]; 800 North Lindbergh Boulevard, St. Louis, MO 63167 (US).

(72) Inventors: FONTAINE, Edward, Armand; 3036 East Kingsfield Road, Pensacola, FL 32514 (US). PARIKH, Sunil, Shantilal; 117 Ridgecrest Drive, Westfield, MA 01085 (US). UDIPI, Kishore; 66 Glenbrook Lane, Longmeadow, MA 01106 (US).

(74) Agent: STANLEY, Howard, Cromwell; Monsanto Company, 800 North Lindbergh Boulevard, St. Louis, MO 63167 (US).

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(54) Title: PROCESS FOR IMPROVING PROPERTIES OF FILLED POLYAMIDES

(57) Abstract

A process for improving the processability in addition to physical properties of filled polyamides is disclosed. The inventive process does not adversely affect the flammability of such filled polyamides, and comprises: wetting a particulate filler, such as carbon black, with a plasticizer in an amount which is from about 60 to about 110 percent of the oil absorption value of the particulate filler; blending the wetted particulated filler with the polyamides; and then molding or extruding the blend into a shaped article.

PROCESS FOR IMPROVING PROPERTIES OF FILLED POLYAMIDES

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention is directed to a process for incorporating particulate fillers such as stabilizing fillers and solid pigments, including, carbon black, into thermoplastic polymers such as polyamides. This invention is also directed to filled polyamide molding compositions and to molded articles prepared therefrom having improved properties.

2. Description of the prior art

The use of stabilizing fillers and solid pigments, such as carbon black, in polyamides is well known. There are certain problems associated with the use of these materials, especially carbon black, in that they may have a tendency to dust, be difficult to handle and to disperse in the polyamides.

U.S.P. 3,945,978 to Berg et al. teaches a process for the production of pourable, pulverulent filler mixtures containing plasticizer oils. Example 1 of the Berg et al. reference shows a process for preparing a carbon black plasticizer oil mixture which is pourable and without any tendency toward coalescing for use in rubbery or elastomeric compositions.

U.S.P. 4,277,288 to Lawrence et al. teaches a process for producing a substantially dry, low dusting, free flowing granular pigment composition comprising (a) contacting a fluidized bed of pigment and a granulating assistant, (b) optionally contacting the granules so obtained with

a surfactant and (c) removing the dry granules from the bed.

However, Berg et al. and Lawrence et al. do not address the problems, i.e. diminishing of certain physical properties, associated with incorporating particulate fillers, such as carbon black, into polyamides.

Polyamide molding and extrusion compositions containing carbon black are disclosed inter alia in U.S.P. 4,391,936 to Marion G. Waggoner and in U.S.P. 4,518,728 to Robert E. Pollard. Waggoner teaches polyamide molding blends containing carbon black and nigrosine and teaches that carbon black can be employed in any of its commercial forms. Waggoner demonstrates, by way of working examples Control D and Control D-1 (displayed in Table IV) that the addition of carbon black (by way of the process disclosed therein) to a nylon 66 base resin results in a molded article having reduced elongation and showing brittle breaks. Pollard teaches using a carbon black that is a concentrate or premix of a 25% by weight dispersion of carbon black in a nylon 6 carrier. Such premixes are preferred over carbon black powder which is difficult to handle and to disperse evenly in the polyamide. The concentrate approach provides an easier method for handling particulate fillers, such as carbon black, but requires separate steps for dispersing the material and preparing the concentrate. This adds to the cost of the final polymer composition. There is a need in the thermoplastic molding and extrusion art for a lower cost, more efficient method for incorporating particulate fillers into thermoplastics without adversely affecting (and possibly improving) the properties of the filled polymers.

SUMMARY OF THE INVENTION

The present invention is therefore directed to a process for improving the processability and tensile strength of, and promoting ductile failure of filled polyamides, while not adversely affecting the flammability of such filled polyamides, which process comprises:

(a) wetting a particulate filler with a plasticizer for the polyamides in an amount which is from about 60 to about 110 percent of the oil absorption value of the particulate filler;

(b) blending the wetted particulate filler with the polyamide; and then

(c) molding or extruding the blend of the wetted particulate filler and polyamide into a shaped article,

where the particulate filler is selected from the group including stabilizing fillers and solid pigments, such as carbon black.

The present invention is also directed to a flame retardant filled polyamide molding composition having improved processability and to a flame retardant, molded article having improved thermal properties, tensile strength and toughness.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The addition of plasticizers to polyamide molding compositions is known to adversely affect the flammability of, in addition to causing a lowering in the heat distortion temperature of molded articles prepared therefrom. By the present invention however, it has been found that by wetting a particulate filler with a quantity of plasticizer prior to blending the filler with a polyamide the heat distortion temperature of molded articles prepared from such filled polyamides is unexpectedly increased, and the flammability of

such articles is surprisingly not adversely affected, while incorporation of the filler into the polyamide is facilitated.

Polyamides which may be employed in the practice of the present invention are commonly known as nylons and include, but are not limited to, nylon 6 or poly(caprolactam), nylon 11 or poly(11-aminoundecanoic acid), nylon 12 or poly(lauryl lactam) or poly(12-aminododecanoic acid), nylon 6,6 or poly(hexamethylene adipamide), nylon 6,9 or poly(hexamethylene azelamide) or poly(hexamethylene noandiamide), nylon 6,10 or poly(hexamethylene sebacamide) or poly(hexamethylene decanediamide), nylon 6,12 or poly(hexamethylene dodecanoamide) or nylon 4 or poly(δ-butyrolactam), nylon 7 or poly(7-aminoheptanoic acid) or poly(7-aminooenanthylic acid), nylon 8 or poly(8-aminocaprylic acid) or poly(8-aminooctanoic acid), nylon 10,6 or poly(decamethylene adipamide) and numerous partially aromatic nylons (PARNs). PARNs result when an aromatic residue or unit is substituted in whole or in part for an aliphatic residue or unit in an aliphatic nylon polymer. For example, substitution of all of the adipic acid [HOOC-$(CH_2)_4$-COOH] residues in nylon 6,6 by those from mixtures of about 50-70% terphthalic acid (TA, or p-HOOC-C_6H_5-COOH)/-50-30% isophthalic acid (IA, or m-HOOC-C_6H_5-COOH) gives suitable PARNs which are high-melting, partly crystalline nylons 6,TA-co-6, IA or poly(hexamethylene tere-coisophthalamides). Other suitable PARNs are partly crystalline nylons 6,6-co-6,-TA, nylons 6,6-co-6,IA, nylons 6,6-co-6,-TA-co-6,IA, and other similar PARNs, including partly crystalline PARNs wherein some or all of the diamine residues have aromatic character and those

containing lactam residues, such as nylons 6-co-6,6-co-6,TA.

Also suitable are various types of copolyamides, block copolymers, and graft copolymers.

Particulate fillers, such as stabilizing fillers and solid pigments, used in the present invention include conventional carbon blacks and other pigments. Such materials typically have particle sizes ranging from about 20 to about 100 nanometers (nm).

Carbon black is usually present in molding compositions in small amounts as a colorant. Carbon black may also be used in higher amounts (e.g., two weight percent loadings) in order to stabilize the resulting molding compositions against ultra-violet radiation by minimizing the absorption of light.

The plasticizers, which are used to wet the particulate fillers, such as carbon black, are those plasticizers conventionally used with polyamides. These plasticizers, which are well known in the art, include phthalates such as diundecylphthalate, dioctyl phthalate, dibutoxyethyl phthalate, butylbenzyl phthalate, dibutyl phthalate, etc.; sulfonamides such as cyclohexyl-p-toluene sulfonamide, N-ethyl-p-toluene sulfonamide, N-ethyl-o,p toluenesulfonamide, o,p Toluene sulfonamide, etc.; trimellitates such as trioctyltrimellitate, tri n-hexyl trimellitate, triisodecyl trimellitate, triisononyl trimellitate, n-octyl-n-decyltrimellitate, etc; epoxidized unsaturated fatty esters; partially hydrogenated terphenols; and other plasticizers used for polyamides.

The oil absorption value for the particulate fillers of the present invention is a

function of the surface and chemical nature of the filler surface and can be readily determined by reference to American Society for Testing and Materials (ASTM) test methods. In particular the oil absorption value for carbon black is readily determined by ASTM D-2414-93 and the oil absorption value for other pigments by ASTM D-1483-84 (Reapproved 1989) and ASTM D-1483-60. Determination of the oil absorption value allows the proper amount of particulate filler and plasticizer to be blended so as to provide a filler that is dry and free flowing but not dusty. Also, as set forth above, the presence of significant amounts of plasticizer may be detrimental to the properties of the molded polyamide article in certain applications. By determining the oil absorption value of the filler and by using an amount of plasticizer that is within about 60 to about 110 percent, and preferably within about 70 to about 90 percent and more preferably within about 75 to about 85 percent of the oil absorption value, one is able to prepare a wetted filler while using an optimum amount of the plasticizer.

The particulate filler of the present invention is preferably wetted with the plasticizer in a powder mixer such as a Henschel mixer. During the preferred wetting process the particulate filler is charged to the mixer, the mixer is turned on to low speed (~ 430 rpm) and the plasticizer introduced into the mixer in finely divided form. Once the plasticizer has been fully introduced the mixer is turned on to high speed (~ 860 rpm) for an additional minute and then stopped. Where overheating during the wetting process can result in clumping of the resultant product it is preferred that cooling water be circulated through the jacket of the mixer throughout the process.

Additionally, it is preferred that high speed mixing be minimized as described above. It is further preferred that process temperatures and the temperature of the wetted product be maintained at or below 45°C.

A preferred polyamide molding composition of the present invention contains:

(1) from about 99 to about 80% by wt. of a polyamide resin;

(2) from about 0.5 to about 10% by wt. of a particulate filler; and

(3) from about 0.5 to about 10% by wt. of a plasticizer,

where the sum of components (1) to (3) total 100% by weight.

A more preferred molding composition comprises:

(1) from about 99 to about 90% by wt. of nylon 6,6;

(2) from about 0.5 to about 5% by wt. of carbon black; and

(3) from about 0.5 to about 5% by wt. of trioctyltrimellitate plasticizer.

The polyamide molding composition of the present invention may also advantageously contain a heat stabilizer component. Such heat stabilizer components preferably contain from about 40 to about 200 ppm copper and more preferably contain from about 50 to about 120 ppm copper.

The present invention is described in more detail with reference to the following working examples which are for the purposes of illustration only and are not to be understood as indicating or implying any limitations on the broad invention described herein.

SPECIFIC EMBODIMENTS

Sample Preparation

A. Formulations

The formulations used in the working examples are set forth in Table I below.

TABLE I

FORMULATIONS TESTED

% BY WEIGHT

Example	A	B	Control C	Control D
Ingredients				
Nylon 6,6	96.00	94.00	91.84	—
Raven C	2.00	2.00	—	—
TOTM	1.84	1.84	—	—
Additive 1	—	—	8.0	—
Additive 2	0.16	0.16	0.16	—
Additive 3	—	2.00	—	—
Additive 4	1800ppm	1800ppm	1800ppm	—

Key:

Nylon 6,6 - used in Examples A, B and C was Vydyne® 21 nylon available from Monsanto Chemical Co., 800 N. Lindbergh Blvd., St. Louis, MO 63167.

Raven C - is carbon black from Columbian Chemicals Co., 1600-T Parkwood Circle, Suite 400, Atlanta, GA 30339 (18 to 20 nanometers (nm) in size).

TOTM - Trioctyltrimellitate plasticizer from Unitex Chemical Corp., 520-T Broome Rd., Greensboro, NC 27406.

Additive 1 - is a 25% carbon black concentrate in nylon obtained from Americhem, Inc., 225 Broad Blvd., East, Cuyahoga Falls, OH 44221.

Additive 2 - is a 25% phthalocyanine blue concentrate in nylon prepared by extruding a blend of 25% phthalocyanine blue and 75% nylon 6,6 (% by wt.).

Additive 3 - is a heat stabilizer package of 84.5% Potassium Iodide, 10.5% Cuprous Iodide, 5% Aluminum Distearate (% by weight).

Additive 4 - is aluminum distearate which is used as an external lubricant.

Control C - is a commercial weather stable black nylon molding composition available from Monsanto Chemical Co. as Vydyne 25

WSP-F. It is the system described in U.S.P. 4,518,728 to Pollard.

Control D - is a commercial weather stable black nylon molding composition available from duPont Chemicals, 1000-T Market St., Wilmington, DE 19898 as Zytel FE-3664.

The oil absorption value for the Raven C carbon black was determined to be 114. The amount of TOTM plasticizer used was 80% of the oil absorption value.

B. Wetting and Preblending

1. Wetting the Carbon Black

45.4 kg of Raven C or carbon black was charged to a 500 liter Henschel mixer. Attached to the underside of the lid of the mixer was a circular (63.5 cm in diameter) metal ring manifold (3.2 cm in diameter) which had about 140 holes (about 1.6 mm in diameter) drilled in. (The large number of holes allowed for the rapid distribution of liquid over a large area inside the mixer which in turn minimized clumping and overheating). The metal ring, secured to the mixer lid by a metal plate bolted to the lid at one of the access holes, was coupled to a large addition funnel via a 6.35 cm in diameter fill pipe. Cooling water was circulated through the jacket of the mixer for the entire process. After the carbon black had been added and the lid secured, the mixer was turned on to low speed (~430 rpm) and 41.9 kg of TOTM was added via the addition funnel. This liquid addition process took about 1 minute. After all the liquid had been added, the mixer was turned off for a few seconds and then turned back on immediately to high speed (~ 860 rpm) for an additional minute and then stopped. The resultant product was a slightly warm (~ 40°C), well dispersed and wetted blend of 52/48 carbon black/TOTM, neither dusty nor sticky. The wetted

carbon black thus prepared was packed in drums for later compounding with the Nylon 6,6.

2. Incorporation of Wet Carbon Black in Nylon 6,6

The Nylon 6,6 and wetted carbon black were physically blended in a Henschel mixer (other additives were also added in this step) at 430 rpm for about 1 minute. The mixture was then placed in a large hopper for subsequent feeding into an extruder. The mixing and blending operation may also be carried out by the tumbling method.

C. Processing

1. Extrusion of the Blend

The mixture of Nylon 6,6 and carbon black was fed into a twinscrew extruder (TSE) using a screw design to provide a high degree of distributive mixing, and extruded under the following conditions:

Set temperature: ~270°C Screw RPM: 250
Stock temperature: ~295°C Feed rate: ~45.5 kg/hr
Vacuum: ~25" Hg

The extruded strands were cooled in a water bath and then pelletized. The pellets were subsequently dusted with an external lubricant (aluminum distearate) and packed into appropriate containers ready for molding. The extrusion process may also be carried out by feeding the wet carbon black and the nylon into the extruder using separate feeders.

2. Molding of the Blend

Molding of the finished product was performed on a Newbury 200 (283 gram barrel capacity) molding machine utilizing a family mold which produced a variety of test specimens. Molding conditions used were more or less standard Nylon 6,6 conditions which include: set temperature about 295°C, mold temperature 93°C, injection time ~ 1 sec, hold and clamp closed time

about 15 seconds each, injection pressure about 5.5 MPa, hold pressure about 4.8 MPa, back pressure about 0.34 MPa, screw rpm about 120. The moisture content of the pellets was in the range of from 0.11 to 0.17%. It is believed that the plasticizing effect of TOTM allows the material to be molded at lower temperatures (275-285°C) with slightly increased injection and hold pressures. The above molding conditions were selected because they produced a good balance of properties and are similar to those conventionally used by molders of nylon 6,6 products.

The specimens to be tested were separated from the rest of the family of parts, allowed to cool, sealed in polyethylene bags to minimize moisture pick up and hermetically sealed in foil lined bags until tested.

Unless otherwise specified, all test specimens were tested on the following day in a "dry as molded" (DAM) condition.

Test Methods

The molded specimens were tested as follows:

(1) Tensile strength at yield and % Elongation at Fail (determined using ten dogbone-shaped specimen bars having nominal cross-sectional dimensions of 0.32 cm thick x 1.27 cm wide, 50 mm/min cross head speed - ASTM Test Method D638-M-89. Measurements in megapascals (MPa) and in %, respectively.

(2) Notched Izod Impact (determined using 0.32 cm thick x 1.27 cm wide x 12.7 cm long specimen bars cut into 6.35 cm long bars to provide two pieces for testing: Gate end (GE)- the piece closest to gate) - ASTM D256-88 using a machined 0.025 cm radius notch in bar. Measurements in Joules per meter (J/m).

(3) Mold Shrinkage (determined using 0.32 cm thick x 1.27 cm wide x 12.7 cm long end gated specimen bars. Specimen bars, sealed upon molding, were equilibrated at 23°C for 24 hours and then the lengths of the bars were measured to within 0.0013 cm and recorded. The difference between the bar length and the length of the mold cavity (12.9134 cm) was then determined for 5 specimen bars and the average used to compute the actual mold shrinkage.) Measurements in cm/cm.

(4) HDTUL or the deflection temperature test (determined using 0.64 cm thick x 1.27 cm wide x 12.7 cm long specimen bars, 1.8 MPa centrally located load, 30°C initial temperature, 2°C/minute temperature rate change) - ASTM Test Method D648-82. Measurements at 0.25 mm deflection in °C.

(5) Spiral Flow (determined by injecting pellets (mold dried to 0.12 ± 0.02%) by way of a VanDorn (168g barrel capacity) injection molding machine, into a mold having a small, fixed cross-

sectional area over an elongated flow path. A moldability index is the length of the elongated flow path that is filled before solidification of the injected molten polymer composition at specified molding conditions, i.e., volumetric injection rate, mold temperature, polymer melt temperature and mold volume. The mold configuration used had a half round profile extended in spiral or serpentine shapes. The moldability index of the molding compositions of this invention is characterized by "Spiral Flow", which is the length of the spirally-elongated, half-round mold that is filled with molten polymer composition under the injection molding conditions specified below. The mold had a flat surface with a round broach having a radius of 2.77 mm, a depth of 2.77 mm and a length of at least 42 cm. A 5 to 15 cm long runner was connected from a 4 mm (diameter) sprue to the mold at the beginning of the spiral. The runner had a cross sectional area of between 2 and 4 times the cross-sectional area of the half round mold.

The injection molding conditions were as follows:

injection pressure:	4.83 MPa
hold pressure:	4.14 MPa
back pressure:	0.21 MPa
barrel temperature:	300°C
mold temperature:	93°C
injection cycle:	
injection time:	2 seconds
hold time:	10 seconds
cooling time:	10 seconds

Shot size was set at 1.25 which provided a cushion of 0.5.

Spiral Flow was measured after equilibrating the molding cycle (e.g., after 30

cycles) and was determined as the average half round injection length of 20 consecutive moldings. A minimum of 30 molded specimens were discarded between samples. The length of spiral flow was measured to the nearest 0.64 cm.

(6) Peak Melting Point, Peak Recrystallization Point, % Crystallinity and Heat of Fusion were derived from differential scanning calorimetry on a Perkin Elmer DSC-7 calorimeter, in an inert atmosphere (nitrogen purged at 20 ml/min) at a heating/cooling rate of 20°C/min. Heat of fusion was measured from the area under the derived melting endotherm and the % crystallinity was based on the heat of fusion value of 188.28 J/g for 100% crystallinity as set forth in 1Journal of Polymer Science, Part A, p. 2697 (1963).

(7) Sperulite Size (determined using approximately 2 micron thick sections cut from the centers of molded specimen bars and examining for sperulite size using a JENAPOL compound polarizing microscope (Model #721021, manufactured by Aus Jena, Jena, Germany, utilizing cross polarized light). Measurements in microns (μ)

(8) Heat Aging - prior to testing for % retention of tensile strength @ yield and % retention of % elongation @ fail molded specimen bars were hung by the gate end grip area in an air circulating oven at 120°C for 1000 hours. The bars were closely inspected for mold flashing prior to each % retention test and any flashes were removed with a flashing tool and/or by light sanding. Mold flashings, when not removed, have been known to cause premature failures. At the end of 1000 hours the specimen bars were removed from the oven and immediately placed on a desiccator to cool for a period of four hours. The specimen bars were then hermetically sealed and thermally equilibrated at

23°C for a period of 24 hours. The heat aged specimen bars were then tested as set forth above for tensile strength at yield and % elongation at fail and the % retention values determined based upon the average value obtained from ten bars.

(9) Flammability per Underwriters Laboratory Test No. UL-94 (vertical burn) - specimens (at specified thicknesses, 0.33 cm, 0.15 cm, 0.08 cm) tested for flammability were supported from the upper end with the longest dimension vertical, by a clamp on a ring stand so that the lower end of the specimen was one centimeter above the top of a burner tube. The burner was then placed remote from the sample, ignited and adjusted to produce a blue flame two centimeters in height.

The test flame was placed centrally under the lower end of the test specimen and allowed to remain for 10 seconds. The test flame was then withdrawn, and the duration of flaming or glowing combustion of the specimen noted. If flaming or glowing combustion of the specimen ceased within 30 seconds after removal of the test flame, the test flame was again placed under the specimen for 10 seconds. The test flame was again withdrawn, and the duration of flaming or glowing combustion of the specimen noted.

If the specimen dropped flaming particles or droplets while burning in this test, these drippings were allowed to fall onto a horizontal layer of cotton fibers (untreated surgical cotton) placed one foot below the test specimen. Significantly flaming particles were considered to be those capable of igniting the cotton fibers.

Materials classified as V-2: did not have any specimens that burned with flaming combustion for more than 30 seconds after either application of the test flame; did not have a total

flaming combustion time exceeding 250 seconds for the 10 flame applications for each set of five specimens; did not have any specimens that burned with flaming or glowing combustion up to the holding clamp; were permitted to have specimens that dripped flaming particles that ignited the surgical cotton; and did not have any specimens with glowing combustion that persisted for more than 60 seconds after the second removal of the test flame.

EXAMPLES A AND B AND CONTROLS C AND D

In Examples A and B and Controls C and D molded test specimens were tested for physical, mechanical, thermal and processing properties. The results are tabulated in Table II.

TABLE II

SUMMARY OF EXAMPLES A AND B AND CONTROLS C AND D

Example	A	B	Control C	Control D
Properties				
Tensile Strength @ Yield (MPa)	82.5	84.1	82.5	79.6
% Elongation @ Fail	36	43	33	41
Brittle Breaks	1	0	1	0
Notched Izod (GE)(J/m)	48.6	48.6	47	56.6
Mold Shrinkage (cm/cm)	0.0168	0.0181	0.0183	0.0208
HDTUL (°C)	74	79.7	72.2	72.7
Spiral Flow, cm @ 300C	73	74.3	76.8	66.7
Peak Melting Point, (°C)	259	259.2	258.6	258.2
Peak Recrystn Point, (°C)	226.6	225.5	223.4	221.4
% Crystallinity	38.7	37.6	37.0	35.1
Heat of Fusion	72.9	70.9	69.7	66.0
Sperulite Size (μ)	3-6	3-6	4-6	4-13
UL-94 Rating 0.33 cm	V-2	V-2	V-2	V-2

Maximum Secs.	9	14	12	4
Total Secs.	41	89	62	14
UL-94 Rating 0.15 cm	V-2	V-2	V-2	V-2
Maximum Secs.	7	6	7	9
Total Secs.	28	29	35	26
UL-94 Rating 0.08 cm	V-2	V-2	V-2	V-2
Maximum Secs.	2	8	4	3
Total Secs.	9	15	9	7
		Heat Aging		
%Retention, Tensile Strength	b.i.g.	106	b.i.g	67
%Retention, Elongation	b.i.g.	96	b.i.g	10

Key:

b.i.g. - broken in gate.

Brittle breaks - were number of breaks (% elongation <15%) per 10 samples tested

It is apparent from the results listed in Table II that the molding compositions of the present invention exhibit improved processability and improved physical properties as compared to the weather stable black nylon molding compositions of Controls C and D. The improved processability of Examples A and B is demonstrated by the peak recrystallization points. The 2 to 5° difference in peak recrystallization points denotes the ability of the present inventive compositions to set faster in the mold. The improved physical properties of Examples A and B are demonstrated by the heat distortion or deflection temperatures, tensile strengths at yield and % elongation at fail values obtained. It is further apparent that the present inventive molding compositions containing a heat stabilizing component demonstrate, upon heat aging, a full retention of tensile strength and elongation, unlike Control D.

In addition, the molding compositions of the present invention exhibit comparable flammability results to those of Controls C and D.

While the preferred embodiments of the present invention have been described, various modifications and substitutions may be made thereto without departing from the spirit and scope of the invention. Accordingly, the present invention has been described by way of illustration, not limitation.

What is claimed is:

CLAIMS

1. A process for improving the processability and tensile strength of, and promoting ductile failure of filled polyamides, while not adversely affecting the flammability of such filled polyamides, wherein the process comprises:

a. wetting a particulate filler, having an oil absorption value, with a plasticizer for the polyamides, in an amount which is from about 60 to about 110 percent of the oil absorption value of the particulate filler;

b. blending the wetted particulate filler with the polyamide; and then

c. molding or extruding the blend of the wetted particulate filler and the polyamide into a shaped article,

wherein the particulate filler is selected from the group including stabilizing fillers and solid pigments, such as carbon black, and mixtures thereof.

2. The process of Claim 1 wherein the particulate filler is wetted with the plasticizer in an amount which is from about 70 to about 90 percent of the oil absorption value of the particulate filler.

3. The process of Claim 1 wherein the particulate filler is carbon black.

4. A process for improving the processability and tensile strength of, and promoting ductile failure of filled polyamides, while not adversely affecting the flammability of such filled polyamides, wherein the process comprises:

a. wetting carbon black, which has an oil absorption value, with a plasticizer for the polyamides, in an amount which is from

about 60 to about 110 percent of the oil absorption value of the carbon black;

b. blending the wetted carbon black with the polyamide; and then

c. molding or extruding the blend of the wetted carbon black and the polyamide into a shaped article.

5. The process of Claim 4 wherein the carbon black is wetted with the plasticizer in an amount which is from about 70 to about 90 percent of the oil absorption value of the carbon black.

6. The process of Claims 1 or 4 wherein the plasticizer is selected from the group consisting of phthalates, sulfonamides, trimellitates, epoxidized unsaturated fatty esters, partially hydrogenated terphenols, and mixtures thereof.

7. The process of Claim 6 wherein the plasticizer is a trimellitate plasticizer selected from the group consisting of trioctyltrimellitate, tri n-hexyl trimellitate, triisodecyl trimellitate, triisononyl trimellitate, n-octyl-n-decyl trimellitate, and mixtures thereof.

8. The process of Claim 7 wherein the plasticizer is trioctyltrimellitate plasticizer.

9. The process of Claims 1 or 4 wherein the polyamide is nylon 6,6.

10. A flame retardant filled polyamide molding composition having improved processability, which comprises: a polyamide resin; a particulate filler having an oil absorption value; and a plasticizer,

wherein the particulate filler is wetted with the plasticizer in an amount which is from about 60 to about 110 percent of the oil absorption value of the particulate filler, prior to adding it to the polyamide, and

wherein the particulate filler is selected from the group including stabilizing fillers and solid pigments, such as carbon black, and mixtures thereof.

11. A flame retardant filled polyamide molding composition having improved processability, which comprises: a polyamide resin; carbon black, which has an oil absorption value; and a plasticizer,

wherein the carbon black is wetted with the plasticizer in an amount which is from about 60 to about 110 percent of the oil absorption value of the carbon black, prior to adding it to the polyamide.

12. The polyamide molding composition of Claim 11 wherein the carbon black is wetted with the plasticizer in an amount which is from about 70 to about 90 percent of the oil absorption value of the carbon black.

13. The polyamide molding composition of Claims 10 or 11 wherein the plasticizer is selected from the group consisting of phthalates, sulfonamides, trimellitates, epoxidized unsaturated fatty esters, partially hydrogenated terphenols, and mixtures thereof.

14. The polyamide molding composition of Claim 13 wherein the plasticizer is a trimellitate plasticizer selected from the group consisting of trioctyltrimellitate, tri n-hexyl trimellitate, triisodecyl trimellitate, triisononyl trimellitate, n-octyl-n-decyl trimellitate, and mixtures thereof.

15. The polyamide molding composition of Claim 14 wherein the plasticizer is trioctyltrimellitate plasticizer.

16. The polyamide molding composition of Claims 10 or 11 wherein the polyamide is nylon 6,6.

17. A flame retardant, molded article having improved thermal properties, tensile strength and toughness and comprising a polyamide molding material comprised of: a polyamide resin; a particulate filler having an oil absorption value; and a plasticizer,

wherein the particulate filler is wetted with the plasticizer in an amount which is from about 60 to about 110 percent of the oil absorption value of the particulate filler, prior to adding it to the polyamide, and

wherein the particulate filler is selected from the group including stabilizing fillers and solid pigments, such as carbon black, and mixtures thereof.

18. A flame retardant, molded article having improved thermal properties, tensile strength and toughness and comprising a polyamide molding material comprised of: a polyamide resin; carbon black, which has an oil absorption value; and a plasticizer,

wherein the carbon black is wetted with the plasticizer in an amount which is from about 60 to about 110 percent of the oil absorption value of the carbon black, prior to adding it to the polyamide.

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(71) Applicant: MONSANTO COMPANY [US/US]; 800 North Lindbergh Boulevard, St. Louis, MO 63167 (US).

(72) Inventors: FONTAINE, Edward, Armand; 3036 East Kingsfield Road, Pensacola, FL 32514 (US). PARIKH, Sunil, Shantilal; 117 Ridgecrest Drive, Westfield, MA 01085 (US). UDIPI, Kishore; 66 Glenbrook Lane, Longmeadow, MA 01106 (US).

(74) Agent: STANLEY, Howard, Cromwell; Monsanto Company, 800 North Lindbergh Boulevard, St. Louis, MO 63167 (US).

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(57) Abstract

A process for improving the processability in addition to physical properties of filled polyamides is disclosed. The inventive process does not adversely affect the flammability of such filled polyamides, and comprises: wetting a particulate filler, such as carbon black, with a plasticizer in an amount which is from about 60 to about 110 percent of the oil absorption value of the particulate filler; blending the wetted particulated filler with the polyamides; and then molding or extruding the blend into a shaped article.

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INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C08K3/04 C08K9/04 C08J3/205 C08J3/20 C08J3/22
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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
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C. DOCUMENTS CONSIDERED TO BE RELEVANT

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European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Leroy, A

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